NASA TECHNICAL MEMORANDUM

UB
NASA TM X-1447

EVALUATION OF TUNGSTEN - URANIUM DIOXIDE COMPOSITES PREPARED BY A TUNGSTEN-VAPOR-CEMENTATION PROCESS

by Charles P. Blankenship

Lewis Research Center
Cleveland, Ohio

NATIONAL AERONAUTICS AND SPACE ADMINISTRATION • WASHINGTON, D. C. • SEPTEMBER 1967

[redacted]

NASA TM X-1447

EVALUATION OF TUNGSTEN - URANIUM DIOXIDE COMPOSITES PREPARED BY A TUNGSTEN-VAPOR-CEMENTATION PROCESS

By Charles P. Blankenship

Lewis Research Center
Cleveland, Ohio

~~RESTRICTED DATA~~

ATOMIC ENERGY ACT OF 1954

GROUP 1
Excluded from automatic downgrading and declassification

[redacted]-TITLE UNCLASSIFIED

This material contains information affecting the national defense of the United States within the meaning of the espionage laws, Title 18, U.S.C., Secs. 793 and 794, the transmission or revelation of which in any manner to an unauthorized person is prohibited by law.

NOTICE

This document should not be returned after it has satisfied your requirements. It may be disposed of in accordance with your local security regulations or the appropriate provisions of the Industrial Security Manual for Safe-Guarding Classified Information.

NATIONAL AERONAUTICS AND SPACE ADMINISTRATION

[redacted]

EVALUATION OF TUNGSTEN - URANIUM DIOXIDE COMPOSITES PREPARED BY A TUNGSTEN-VAPOR-CEMENTATION PROCESS (U)

by Charles P. Blankenship

Lewis Research Center

SUMMARY

Development of a vapor-cementation process for fabrication of tungsten - 20-volume-percent uranium dioxide (UO_2) composite cylinders was sponsored by NASA in conjunction with the tungsten water-moderated nuclear-rocket reactor concept. Tungsten-coated UO_2 particles were infiltrated and bonded together in plate and cylinder geometries by vapor deposition of tungsten. Representative samples prepared by the cementation process were evaluated to determine the status of the fabrication process with respect to composite density, chemical purity, dimensional uniformity, and fuel distribution. In addition, attempts were made to determine the high-temperature (2500° C) fuel-retention characteristics and mechanical properties of the cemented composites.

The cemented composites met the goals of complete encapsulation of the UO_2, homogeneous fuel distribution, and dimensional uniformity. However, composite densities, which ranged from 94 to 96 percent of theoretical, were less than the goal of 98 percent of theoretical. Also, the composites did not have the desired chemical purity, with total halogen and carbon contents greater than 25 parts per million each.

All the vapor-cemented composites were extremely fragile. Specimens subjected to a high-temperature tensile test fractured at the coated-particle interfaces at relatively low stresses compared with a reference material fabricated by hot roll compaction of tungsten-coated UO_2 particles.

Under static and cyclic heating conditions to 2500° C, fuel losses were excessively high in composites believed to be contaminated by tungsten oxyfluorides. The composite sample having the lowest total halogen content and presumed to be free of oxyfluorides exhibited better cyclic fuel-retention characteristics than the reference material and did not exhibit the severe matrix deterioration usually found in thermally cycled tungsten-UO_2 composites. Matrix grain-size stability in conjunction with higher purity probably accounted for the relatively good performance of this sample.

Further development is required to improve the density, the fuel-retention capability, and the mechanical strength before cemented tungsten-UO_2 composites can be considered for use in reactor fuel elements.

[redacted]

INTRODUCTION

The tungsten water-moderated nuclear-rocket reactor concept (TWMR), studied at the NASA Lewis Research Center (ref. 1), uses a dispersion-type fuel element of uranium dioxide (UO_2) particles in a continuous tungsten matrix. A concentric cylinder fuel-element configuration was considered for the proposed reactor because of the large surface area available for heat transfer to the hydrogen propellant. The proposed fuel-element configuration contained 10 concentric cylinders each with a wall thickness of 0.5 millimeter with the largest cylinder diameter being 51 millimeters. A 1.5-millimeter gap between successive cylinders allowed for passage of the hydrogen. Fuel loadings ranged from 10- to 30-volume-percent UO_2, and fuel losses were required to be less than 1 percent after twenty-five 10-minute cycles to 2500° C in hydrogen. Correspondingly, static losses should be less than 1 percent after an equivalent time at temperature.

Several methods of fabricating seamless, tungsten-UO_2 cylinders for this application have been investigated, including hot extrusion of composite tubing (ref. 2), magnetic-pulse swaging (ref. 3), and vapor cementation (ref. 4). Excessive elongation of the UO_2 appears to be a major drawback to hot extrusion, while sufficient energy was not available in the magnetic-pulse swaging process to densify the tungsten, even at 1400° C. Vapor cementation (which consolidates tungsten-coated UO_2 particles by vapor infiltration of tungsten) was considered since mechanical working is not required and since cementation could be accomplished at relatively low temperatures (<500° C). Furthermore, detrimental effects associated with the greater thermal expansion of the dispersed UO_2 could possibly be minimized by using the cementation process. For example, UO_2 spheres with internal voids could be encapsulated in tungsten and formed into a solid body by cementation without affecting the density of the UO_2. On subsequent heating to higher temperatures, some of the volume change associated with thermal expansion would be taken up by the voids in the UO_2. The use of tungsten-coated particles assures complete encapsulation of the fuel and a more homogeneous fuel distribution compared with conventional powder-metallurgy methods of incorporating uncoated UO_2 particles in a tungsten matrix.

Development of a tungsten-vapor-cementation process was studied by the Nuclear Materials and Equipment Corporation (NUMEC) under an NASA-sponsored contract (ref. 4). The objective of that study was to demonstrate the feasibility of fabricating 38-millimeter long, thin-wall (~0.5 mm) tungsten-UO_2 cylinders with diameters of 13, 32, or 51 millimeters. Although fuel loadings of 10 to 30 volume percent are required in fueled cylinders for the TWMR, a nominal 20-volume-percent loading was used in this development program. The following goals were established for this cementation process:

(1) Composite density greater than 98 percent of theoretical

(2) Complete encapsulation of the UO_2

[redacted]

(3) Homogeneous fuel distribution

(4) Uniform wall thickness (±0.025 mm)

(5) Chemical purity greater than 99.9 percent with total halide and carbon content each less than 25 parts per million

In addition to the cylinders, tungsten - 20-volume-percent UO_2 plates (25 by 150 by 0.5 mm) were fabricated by using similar processing techniques.

Representative samples prepared by the cementation process were evaluated at Lewis, and the results of this evaluation are described herein. The samples (primarily from plates) were examined for density variations, chemical purity, dimensional uniformity, and microstructure in determining the status of this fabrication process with respect to the goals. In addition, attempts were made to determine the high-temperature (2500° C) fuel-retention characteristics and mechanical properties of the cemented composites.

Where possible, test results from the cemented composites are compared with similar properties obtained from tungsten-UO_2 composites fabricated by hot roll compaction of tungsten-coated UO_2 particles (ref. 5), a process in a more advanced state of development.

FABRICATION DEVELOPMENT

The vapor-cementation processes used to fabricate the specimens evaluated in this study are described in detail in reference 4 and are summarized in this section. Specimen consolidation was accomplished by tungsten cementation (by the hydrogen reduction of tungsten hexafluoride) of tungsten-coated UO_2 microspheres containing approximately 90 percent dense, 30- to 60-micron-diameter UO_2. After coating, the spheres were approximately 75 microns in diameter. Details concerning the preparation of tungsten-coated UO_2 particles are described in reference 5. The coated particles were contained in the desired geometry by a mold, and tungsten was deposited on the particle surfaces as the gaseous reactants passed through the voids between the particles. Two methods (termed "forced-flow" and "free-diffusion" cementation) were studied for passing the reactants through a body of spherical particles. Processing parameters studied included reactant flow rates, reactant concentrations, cementation temperature and time, and mold design.

Tungsten-UO_2 cylinders were fabricated by a two-step cementation process. Tungsten-coated UO_2 spheres, contained in a cylindrical cavity of brass or copper, were cemented to form a free-standing body by forcing the reactants between the spheres in the axial direction (forced-flow cementation). Deposition of tungsten on the surfaces of the spherical particles (by reduction of the halide) gradually reduced the volume between

adjacent spheres until they were cemented together. Densities near 85 percent of theoretical were obtained. Final densification was accomplished by surface infiltration allowing free diffusion of the reactant gases into the exposed voids. In both steps, cementation temperatures ranged from 225° to 350° C. Since most of the development efforts were directed toward fabricating cylinders with densities greater than 98 percent of theoretical, cementation parameters were not optimized for all three cylinders diameters.

Plates (25 by 150 by 0.5 mm) were fabricated by placing the coated spheres in a Zircalloy-2 mold with one face exposed. Cementation was accomplished by free diffusion, that is, the reactants were passed over the exposed surface (25 by 150 mm) and allowed to diffuse throughout the body of spheres. Cementation temperatures ranged from 180° to 225° C.

In both fabrication processes, the cementation temperatures were relatively low compared with normal tungsten-deposition temperatures (>500° C). The lower temperatures result in an inefficient process, but they prevent rapid tungsten depositon, which would have a greater tendency to fill particle voids near the surface prior to complete infiltration. After cementation, all the composites were heat treated in hydrogen for 1 hour at 1300° C to eliminate the bond interfaces between the coated particles and the infiltrated matrix. Representative plate and cylinder samples fabricated by the cementation process are shown in figure 1.

EVALUATION PROCEDURE

Most of the evaluation study was conducted on plate samples since comparative data were available from plates fabricated by hot roll compaction of tungsten-coated UO_2 particles. The latter composite is referred to as the reference material. Other than density and general microstructure, no attempt was made to determine if the differences in cylinder and plate cementation parameters had any effect on composite properties. The plate samples received for evaluation were examined radiographically to determine the uniformity of tungsten infiltration. Density measurements were made of selected plate and cylinder samples by both water- and mercury-displacement techniques. Samples were chemically analyzed for carbon and halogen impurities, which are believed to be detrimental to fuel-retention properties. Chemical analyses of the tungsten-coated UO_2 particles used to fabricate the specimens were furnished by the contractor (NUMEC). The plate samples were analyzed by an independent laboratory using the procedures described in reference 6.

High-temperature fuel-retention properties of selected specimens were determined in both cyclic and static (isothermal) heating conditions by the procedure described in reference 7. In the thermal cycling tests, specimens (35 by 25 by 0.5 mm) cut from the

plate samples were heated in flowing hydrogen (1 standard m^3/hr) from ambient temperature to 2500° C in 2 minutes, were held at temperature for 10 minutes, and were cooled to ambient temperature in 2 minutes. Specimen weight losses were determined after every 5 cycles with most of the specimens subjected to a total of 25 cycles. Static fuel losses were determined by continually heating similar size specimens at 2500° C in flowing hydrogen (1 standard m^3/hr) for 2 hours. In all cases, the decrease in specimen weight was assumed to be entirely UO_2 loss. Tensile strengths of the composites were determined at 2200° and 2500° C in vacuum (~ 1 mN/m^2) at a crosshead speed of 0.76 millimeter per minute by using the procedure described in reference 8. The samples used for this study and the tests to which they were subjected are summarized in table I.

RESULTS AND DISCUSSION

General Observations

The composite cylinders were uniform, and wall thicknesses were within the goal of ±0.025 millimeter even with the relatively thick cladding (0.13 to 0.20 mm, fig. 2) from overexposure during free-diffusion cementation. Because of the cladding, the measured densities of the cylinders were not representative of a tungsten - 20-volume-percent UO_2 composite. Density measurements of selected specimens from the plate samples, which did not have a thick cladding, ranged from 94 to 96 percent of theoretical; however, radiographic analysis indicated slight density variations along the plate lengths. From the metallographic examination, there appeared to be no difference in matrix density between the cylinders and plates, both having relatively large voids at some of the coated particle interstices (figs. 2 and 3). These void areas were characteristic of the cemented composites and resulted from particle-to-particle bridgement prior to complete tungsten infiltration. For both plates and cylinders, additional parameter studies are required to achieve higher densities.

Fuel distribution appeared to be homogeneous in the cemented composites and was considered to be within the program goal. Complete encapsulation of the UO_2 was accomplished by the use of tungsten-coated UO_2 particles. Although the chemical purity of the composites was 99.9 percent or greater (ref. 4), total halogen and carbon contents were greater than the goal of less than 25 parts per million each (table II). Part of the halogen contaminant was present in the coated particles, and some was entrapped during cementation. Most of the carbon was concentrated in the coated particles. After cementation, carbon contents near 40 parts per million were reported (ref. 4).

Post deposition heat treating at 1300° C for 1 hour did not eliminate completely the bond interfaces between the coated particles and the infiltrated tungsten, as shown in

figure 4. This observation is not in accord with earlier experience in the contractor's program in which a 1300° C heat treatment for 1/2 hour eliminated bond interfaces. The difference is thought to result from the higher purity tungsten hexafluoride used in the early part of the program. Even though inhibited by impurities, grain growth did occur in the cemented composites at relatively low temperatures. The driving force for grain growth is probably related to grain misorientation (ref. 9) between the tungsten coating and the infiltrated matrix.

The cemented composites were extremely fragile as a result of the relatively large voids in the matrix and possibly weak interparticle bonding.

Mechanical Properties

Since the plate specimens were extremely fragile, tensile-strength data were obtained from only two samples (NU-2 and NU-8). The other four specimens prepared for testing (table I) broke either during attachment of the thermocouple or during assembly in the tensile grips. Normally, this problem is not encountered with the more dense, roll-compacted tungsten-UO_2 composites (reference material).

At 2200° C, specimen NU-2 exhibited a tensile strength of 18.6 meganewtons per square meter (2700 psi), while at 2500° C specimen NU-8 had a tensile strength of only 6.2 meganewtons per square meter (880 psi). In comparison, the reference material has a tensile strength of 31.7 and 18.6 meganewtons per square meter (4600 and 2700 psi) at temperatures of 2200° and 2500° C, respectively. Metallographic examination indicated that the cemented composites fractured near the coated-particle interfaces (fig. 5). The weakness of the cemented bonds probably resulted from a combination of notch effects associated with the relatively large voids present at some of the particle interstices and with the presence of impurities as evidenced by porosity at the coated-particle interfaces. This type of porosity in tungsten-UO_2 composites has been attributed to substrate (coated particle) surface impurities (ref. 10).

Fuel-Retention Properties

Static tests. - Under static test conditions, the cemented composites exhibited excessively high fuel losses, 18- to 40-weight-percent UO_2 in 2 hours at 2500° C (table III). Fuel loss under these conditions has been attributed to surface vaporization of the UO_2 at elevated temperatures (ref. 7). Factors that may accelerate this type of fuel loss and that were characteristic of the cemented composites evaluated include the presence of certain impurities (primarily halides and carbon) and matrix voids. Impurities that may

have contributed to the high fuel losses include those believed to have been introduced from the tungsten hexafluoride used in the cementation process (i. e., tungsten oxyfluorides, refs. 4 and 5). The chemical analyses data listed in table II indicate that both carbon and fluorine, as well as other halogens, were present in quantities greater than that listed in the original goals and in quantities believed sufficient to promote fuel loss. Although the chemical interactions and mechanisms promoting fuel loss have not been defined, the impurities probably caused the loss of particle-coating integrity in the cemented composites, as shown in figure 6(a). Furthermore, increased amounts of internal porosity near the coated-particle interfaces after static testing suggests that either the impurities or their reaction products segregated in these areas and were volatilized at the high test temperature. Certain unidentified trace impurities believed to be associated with halides, oxyhalides, and possibly carbon are known to produce grain-boundary porosity in vapor deposited tungsten even in the absence of UO_2 (ref. 10). Thus, the high fuel losses encountered during static testing probably resulted from a combination of (1) the loss of coated particle integrity, (2) porosity generated at high temperatures and located near the coated-particle interfaces, and (3) matrix voids present after fabrication, all of which created open paths through the tungsten matrices to enhance the loss of UO_2 by vaporization. The open paths could lead to the surface of the specimens or to the edges exposed during sectioning the specimens from the plates.

In comparison, static fuel losses from the roll-compacted, reference material were less than 1 percent after 2 hours at 2500° C, and this material showed no porosity after testing (fig. 6(b)). The fact that the reference material contained nearly twice as much fluorine as the cemented composites (table II) suggests that the form or distribution of fluorine contamination may be more detrimental to fuel-retention properties than the total amount present. In the cemented composites, fluorine contamination was probably in the form of oxyfluorides. In the roll-compacted material, the nature of the halide contamination was unknown. However, the temperature of consolidation for the reference material (1700° to 1900° C) probably yielded a more homogeneous halide distribution; whereas, halide contamination in the cemented composites appeared to be localized near the coated-particle interfaces. In addition, there may have been differences in the quality of the coated particles used in fabricating the two composites.

Thermal cyclic tests. - Accelerated rates of fuel loss from tungsten-UO_2 composites under thermal cyclic conditions have been attributed to partial UO_2 decomposition at elevated temperatures followed by the precipitation and migration of free uranium at lower temperatures (ref. 7). Free uranium diffusing through the matrix grain boundaries eventually destroys composite integrity and results in the rapid loss of UO_2 by direct vaporization. This type of behavior as exhibited by the reference material is shown graphically in figure 7. The rate of fuel loss increases rapidly after approximately five

10-minute cycles. In addition to causing matrix porosity, impurities such as carbon and the halogens are believed to be detrimental to cyclic fuel-retention properties of tungsten-UO_2 composites by promoting the decomposition of UO_2.

The cyclic fuel-loss characteristics of the cemented composite samples NU-3, NU-9, NU-11, NU-12, NU-14, and NU-15 were variable, and the total losses were excessively high. The range of fuel losses for the group was within the band shown in figure 7. Metallographic examination showed that the cycled specimens exhibited porosity near the coated-particle interfaces and, in general, loss of coated-particle integrity (fig. 8(a)) similar to specimens tested statically (fig. 6(a)). The poor performance of these specimens is probably associated with the presence of oxyfluorides (all specimens except NU-12 had total halogen contents greater than 220 ppm).

Samples NU-1 and NU-1A, prepared early in the program with tungsten hexafluoride believed to be free of oxyfluorides, had the lowest total halogen content of the specimens tested. Specimen NU-1A cracked in several sections after five cycles which rapidly increased the fuel loss rate by vaporization of the exposed UO_2. Test results from two NU-1 specimens showed a relatively low and constant rate of UO_2 loss (fig. 7). After 15 cycles, the total fuel loss was less than any of the other specimens tested including the reference material. Since sample NU-1 exhibited much lower losses than the other cemented composites, it was subjected to additional cyclic tests. The rate of UO_2 loss remained constant, and the total loss after 80 cycles was 62 percent. Metallographic examination of NU-1 showed large, unfilled voids typical of the cemented composites but relatively small amounts of fine internal porosity at the coated-particle interfaces. The porosity did not increase appreciably from 25 to 80 cycles (figs. 8(b) and (c), respectively), and the specimens did not show the loss of particle-coating integrity found in the other cemented composites. The better performance of NU-1 is probably related to higher purity and possibly the absence of oxyfluorides.

Although the total fuel losses from sample NU-1 were relatively high and well above the reactor fuel-element goal (less than 1 percent loss after 25 cycles to 2500° C), the specimens did not exhibit the severe matrix deterioration usually found in thermally cycled tungsten-UO_2 composites (ref. 7) including the reference material. Similar behavior has been obtained with composites having metal oxide stabilizers in the fuel or matrix or with very fine (<1 μm) UO_2 particles (refs. 11 and 12). However, the results for specimen NU-1 are the first observation of relatively good resistance to thermal cycling in unstabilized tungsten-UO_2 composites with relatively large fuel particles. The unusual performance of sample NU-1 is probably related to the combination of a higher purity material and the relatively small grain size of the tungsten matrix, which was maintained during thermal cycling. The grain size of NU-1 after 25 cycles was approximately one-tenth that of the reference material exposed to only 10 cycles (figs. 9(a) and (b)). In addition, specimen NU-1 did not show an appreciable increase in grain size after

80 cycles (fig. 9(c)). In inhibiting fuel losses, fine-grain matrices possibly reduce deleterious UO_2 reactions with grain boundary impurities because of the lower impurity concentration per unit area. Also, fine-grain matrices provide a more tortuous path for fuel migration to the surface and may be less susceptible to matrix deterioration by grain-boundary separation.

The resistance to matrix grain growth in the strain-free, cemented composites is probably related to grain-boundary pinning by segregated impurities. This condition appears to occur once a nearly equiaxed structure has been established. Matrix voids, as well as impurities, may aid in maintaining a fine grain structure. Since a fine-grain matrix was characteristic of all thermally cycled cemented composites (figs. 9(a) and (d)), the difference in fuel-loss behavior suggests that some impurity content is desirable for inhibiting grain growth, but an excessive amount, particularly in the form of oxyfluorides, may accelerate fuel loss even in a fine-grain matrix. Further studies are required to substantiate this hypothesis and to determine an optimum impurity concentration.

SUMMARY OF RESULTS

Vapor-cemented, tungsten - 20-volume-percent uranium dioxide composites met the initially established goals of complete encapsulation of uranium dioxide, homogeneous fuel distribution, and uniform wall thickness. The composites did not meet the density and chemical purity goals. Further development efforts are required to improve the density, the fuel-retention capability, and the mechanical strength of cemented composites before they can be considered for reactor fuel elements. Specific results of the evaluation are as follows:

1. Cemented-composite densities ranged from 94 to 96 percent of theoretical. The relatively low densities resulted from particle-to-particle bridgement prior to complete tungsten infiltration.

2. The chemical purity of the composites was 99.9 percent or greater with total halogen contents ranging from 124 to nearly 300 parts per million.

3. The composite cylinders were uniform and wall thicknesses were within ±0.025 millimeter.

4. Fuel distribution in the cemented composites was homogeneous and considered to be within the program goal.

5. All the vapor-cemented composites evaluated were extremely fragile. Specimens subjected to a high-temperature tensile test fractured at the coated particle interfaces at relatively low stresses compared with a reference material fabricated by hot roll compaction of tungsten-coated uranium dioxide particles.

6. Under both static and cyclic heating conditions to 2500° C, fuel losses from composites believed to be contaminated by oxyfluorides were excessively high.

7. The composite sample having the lowest total halogen content (NU-1) and presumed to be free of oxyfluorides exhibited better cyclic fuel-retention characteristics than the reference material. In addition, the microstructure did not exhibit the severe matrix deterioration usually found in thermally cycled tungsten - uranium dioxide composites. Grain-size stability in conjunction with higher purity probably accounted for the relatively good performance of this sample.

8. The matrix grain size of the cemented composites exhibited a high degree of stability at elevated temperatures. The resistance to grain growth in the strain-free, cemented composites is probably related to grain boundary pinning by segregated impurities.

Lewis Research Center,
National Aeronautics and Space Administration,
Cleveland, Ohio, April 5, 1967,
122-28-01-01-22.

REFERENCES

1. Lietzke, Armin F.: Water-Moderated Tungsten Nuclear Rocket Reactor Concept. NASA TM X-1044, 1965.

2. Blankenship, Charles P; and Gyorgak, Charles A.: Preliminary Investigation of Extruding Composite Tungsten - Uranium Dioxide Tubing. NASA TM X-1072, 1965.

3. Abraham, L.; Katz, N. H.; and Supan, E. C.: Development of a Magneforming Process for the Fabrication of Thin-Wall Tungsten Cylinders. Rep. No. AI-65-12 (NASA CR-54316), Atomics International, Jan. 13, 1965.

4. Nyce, A. C.; Burke, D. F.; and Vondra, B. L.: The Fabrication of Tungsten - Uranium Dioxide Composite Cylinders by the Tungsten Cementation Process. Rep. No. NUMEC-3728 (NASA CR-54315), Nuclear Materials and Equipment Corp., Oct. 1966.

5. Watson, Gordon K; Caves, Robert M.; and Saunders, Neal T.: Preparation and Roll Compaction of Tungsten-Coated Uranium Dioxide Particles. NASA TM X-1448, 1967.

6. Weber, C. W.; and Kwasnoski, T.: Determination of Impurities in Tungsten - Uranium Dioxide Mixtures. Rep. No. K-1621, Rev. (NASA CR-54275), Union Carbide Nuclear Co., Feb. 19, 1965.

7. Gedwill, Michael A.; Sikora, Paul F.; and Caves, Robert M.: Fuel-Retention Properties of Tungsten - Uranium Dioxide Composites. NASA TM X-1059, 1965.

8. Buzzard, Robert J.; and Gill, Frank L.: High-Temperature Mechanical Properties of a Tungsten - Uranium Dioxide Composite. NASA TM X-1018, 1964.

9. Weinberg, A. F.; Lindgren, J. R.; Elsner, N. B.; and Mills, R. G.: Suppression of Grain Growth in Vapor-Deposited Tungsten up to 2500° C. Nucl. Appl., vol. 1, no. 6, Dec. 1965, pp. 581-583.

10. Caves, Robert M.: Evaluation of Vapor Deposited Tungsten Claddings on Tungsten - Uranium Dioxide Composites. NASA TM X-1449, 1967.

11. Gluyas, Richard E.; and Gedwill, Michael A.: Stabilization of Tungsten - Uranium Dioxide Composites Under Thermal Cycling Conditions. NASA TM X-1295, 1966.

12. Gedwill, Michael A.: An Investigation of Some Variables Affecting and Methods of Inhibiting Thermal-Cyclic Fuel Losses From Tungsten - Uranium Dioxide Composites. NASA TM X-1296, 1966.

TABLE I. - EVALUATION TESTS OF TUNGSTEN - 20-VOLUME-PERCENT UO_2 PLATE SPECIMENS

Specimen		Test		
NASA identity	NUMEC[a] experiment numbers	Tensile strength	Static fuel loss	Cyclic fuel loss
NU-1	23			×
NU-1A	24			×
NU-2	43	×		
NU-3	44	×		×
NU-8	62 and 64	×		
NU-9	54 and 61		×	×
NU-10	56	×		
NU-11	47 and 61		×	×
NU-12	62 and 64	×		×
NU-13	56	×		
NU-14	51 and 58		×	×
NU-15	52 and 58			×

[a]See ref. 4 for cementation data.

TABLE II. - CHEMICAL ANALYSES OF COATED PARTICLES AND REPRESENTATIVE PLATE SAMPLES

Sample identity	Analysis, ppm					Total halogen content
	Coated particles[a]			Plate samples[b]		
	Carbon	Fluorine	Chlorine	Fluorine	Other halogens[c]	
NU-1	154	15	--	73	51	124
NU-1A			--	[d]155	[d]39	194
NU-2			--	240	34	274
NU-3			--	[d]215	[d]38	253
NU-8[e]	15	<5	50	147	12	159
NU-9	154	15	--	[d]175	[d]57	232
NU-11	154	15	--	[d]220	[d]68	288
NU-12[e]	15	<5	50	126	18	144
NU-14	154	15	--	209	52	261
NU-15	154	15	--	[d]180	[d]57	237
Reference material[b]	15	450	11	390	---	≥390

[a]Analysis by NUMEC.

[b]Analysis conducted by commercial laboratory.

[c]Reported as chlorine.

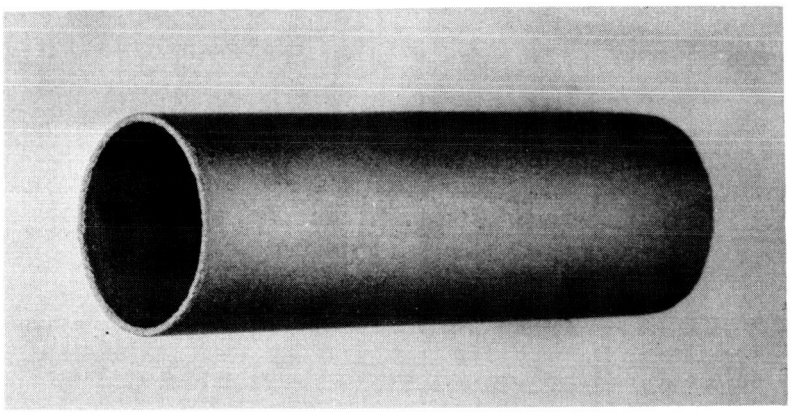
[d]Average of duplicate analyses.

[e]Initial particle coating by hydrogen reduction of tungsten hexachloride (~5 μm), remainder by hydrogen reduction of tungsten hexafluoride.

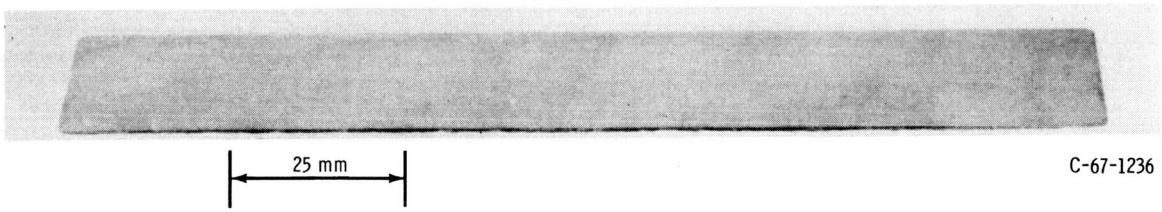
TABLE III. - RESULTS OF STATIC FUEL-RETENTION TESTS FOR TUNGSTEN - 20-VOLUME-PERCENT UO_2 COMPOSITES

[Test conditions, 2 hr at 2500° C in flowing hydrogen.]

Specimen	UO_2 loss, wt. %
NU-9	18.4
NU-11	25.0
NU-14	39.7
Reference material	<1.0



(a) Tungsten-UO_2 cylinder, 13 millimeters in diameter.



(b) Tungsten-UO_2 plate, 150 by 25 by 0.5 millimeters.

Figure 1. - Representative tungsten - 20-volume-percent uranium dioxide cylinder and plate samples fabricated by vapor-cementation.

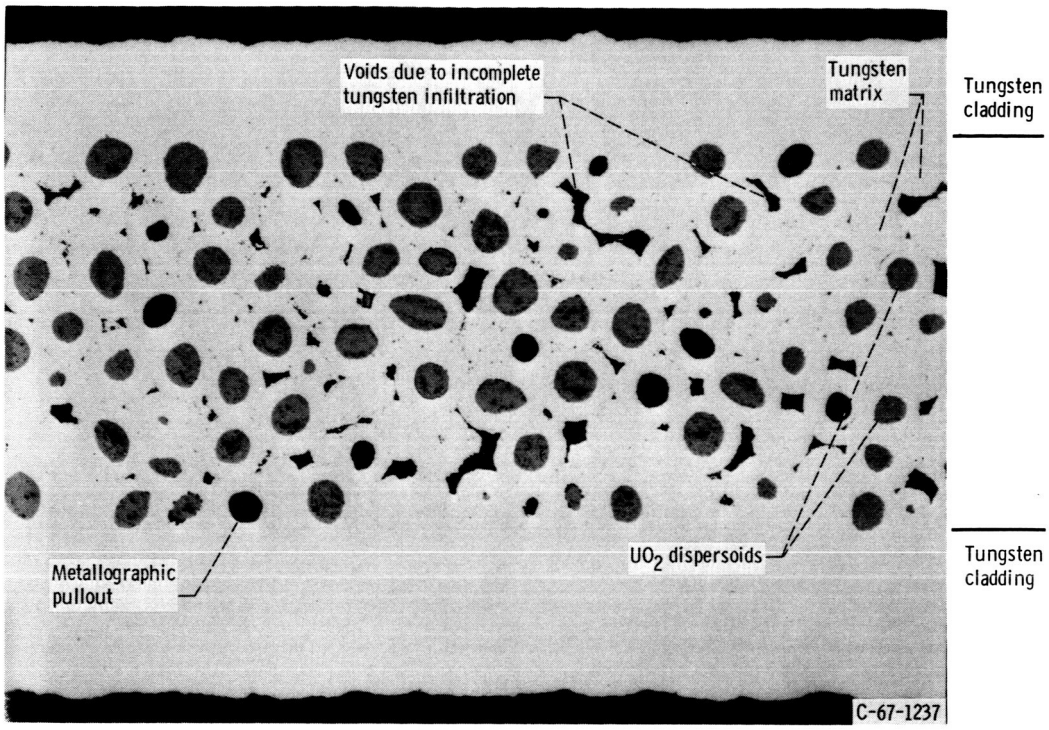


Figure 2. - Typical microstructure of tungsten - 20-volume-percent uranium dioxide composite cylinder prepared by vapor-cementation. Longitudinal section; unetched. X100.

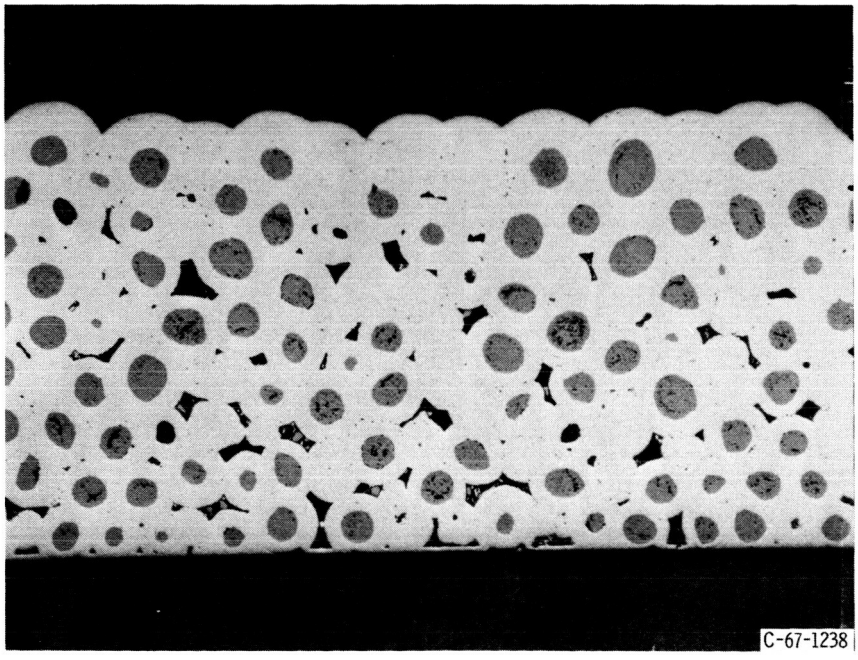


Figure 3. - Typical microstructure of tungsten - 20-volume-percent uranium dioxide composite plate prepared by vapor-cementation. Unetched. X100.

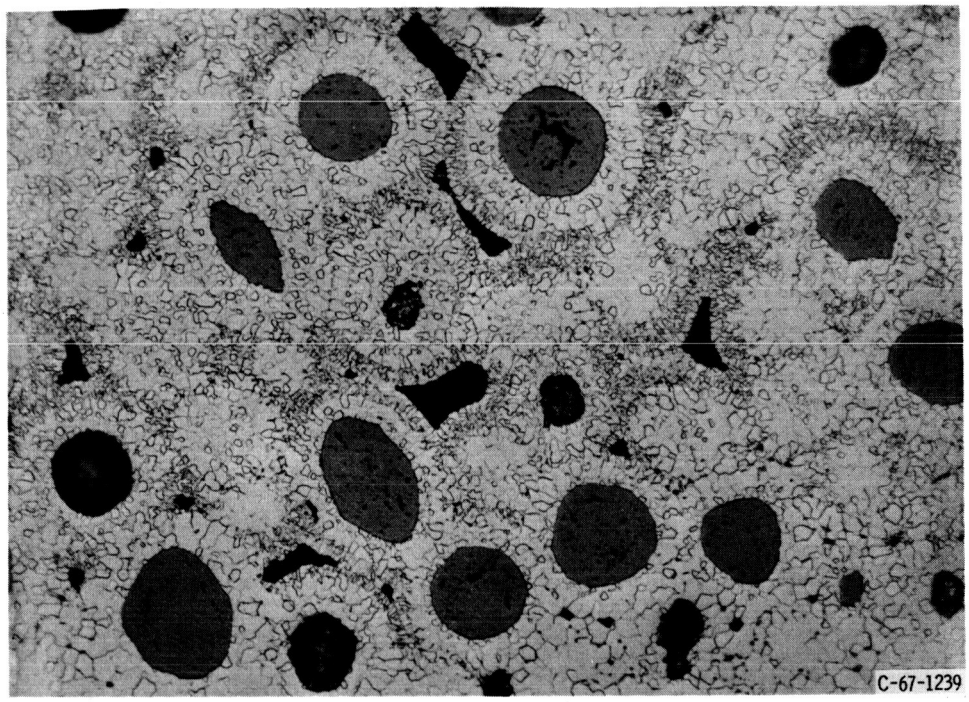


Figure 4. - Typical microstructure of vapor-cemented composites. Etchant, Murakami's reagent. X250.

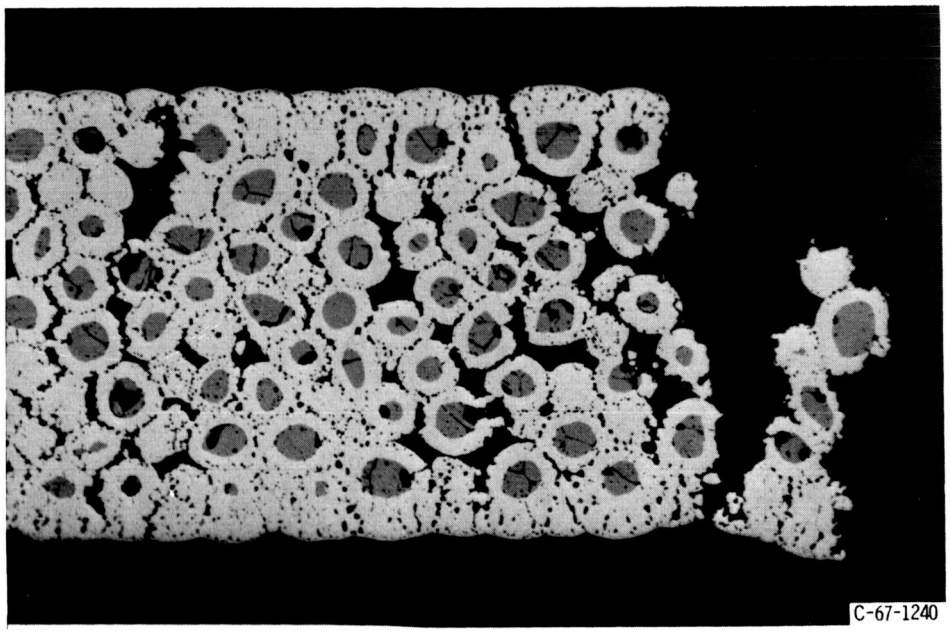
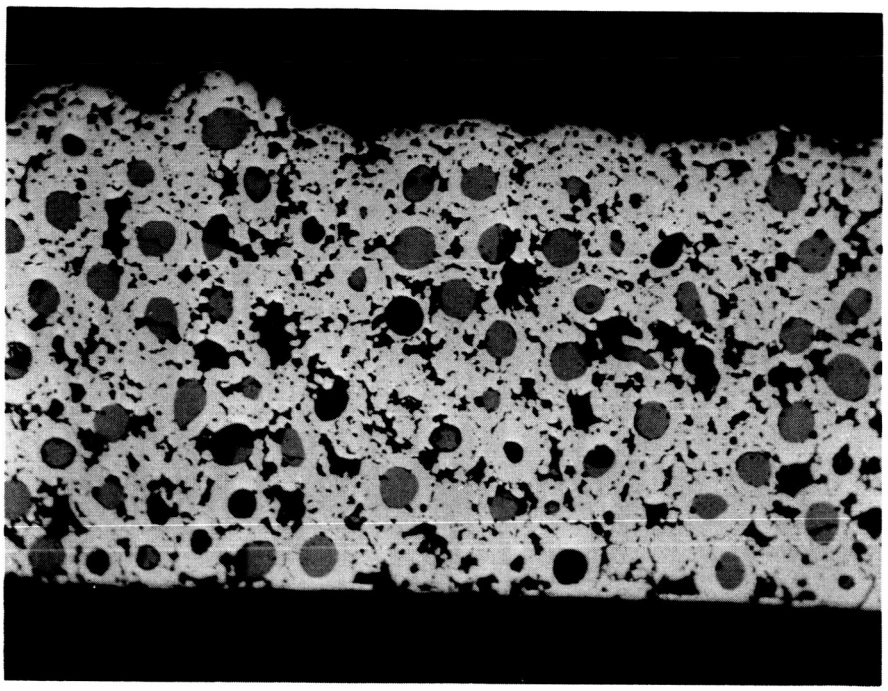
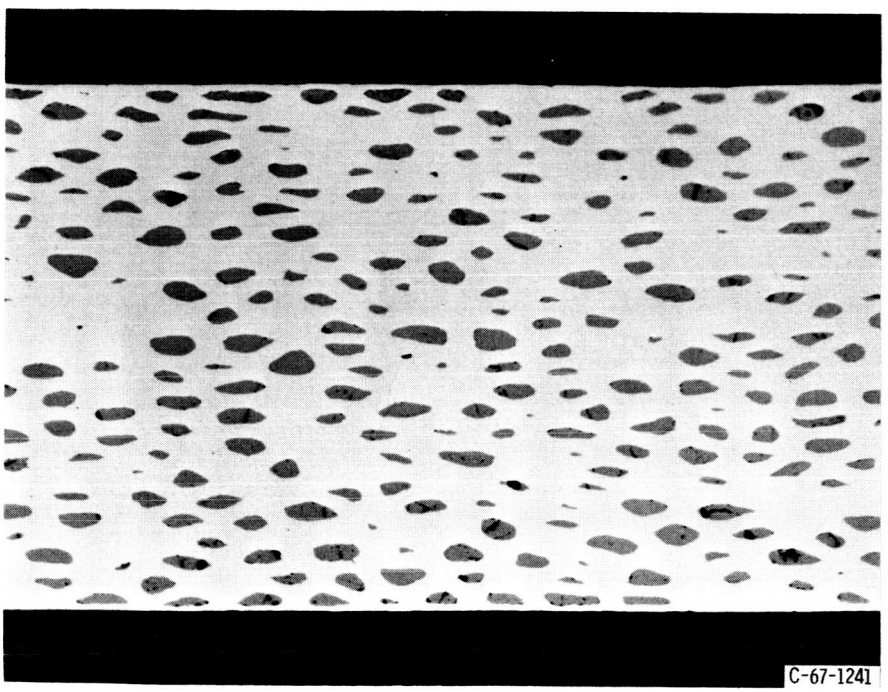


Figure 5. - Tensile fracture of vapor-cemented, tungsten - 20-volume-percent uranium dioxide composite tested at 2500° C. Unetched. X100.



(a) Vapor-cemented composite.



(b) Roll-compacted, reference material.

Figure 6. - Representative microstructures of tungsten - 20-volume-percent uranium dioxide composites after static testing for 2 hours in flowing hydrogen at 2500° C. Unetched. X100.

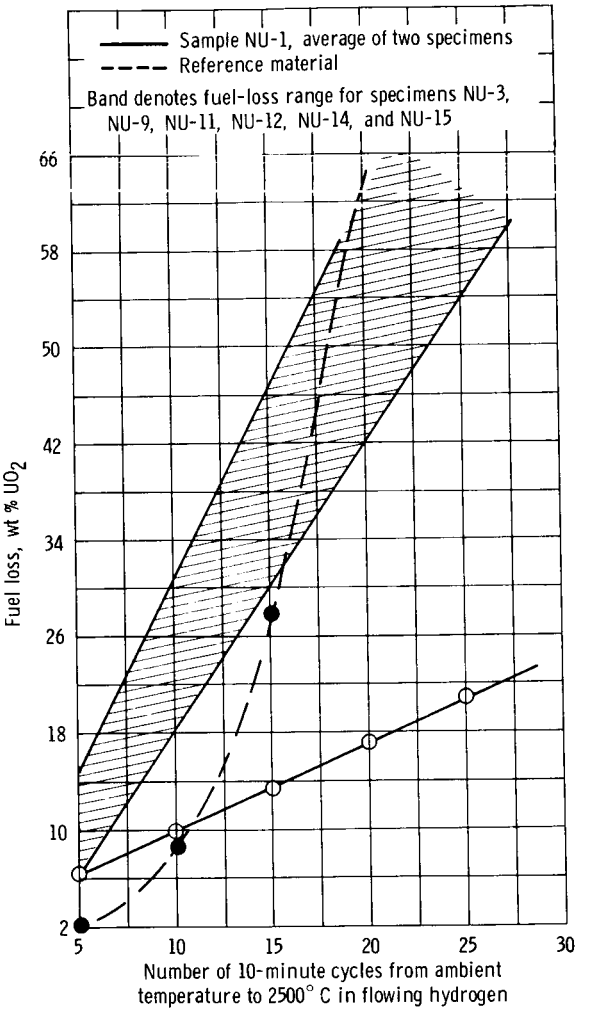
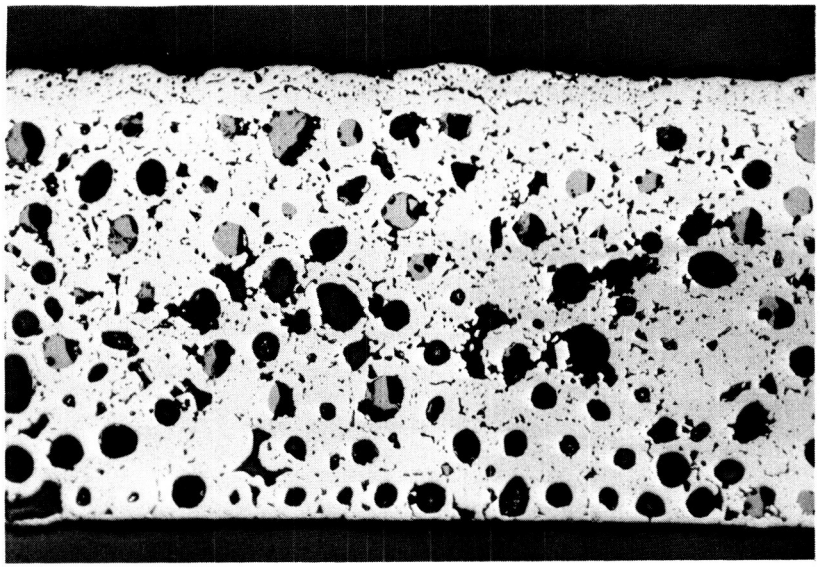
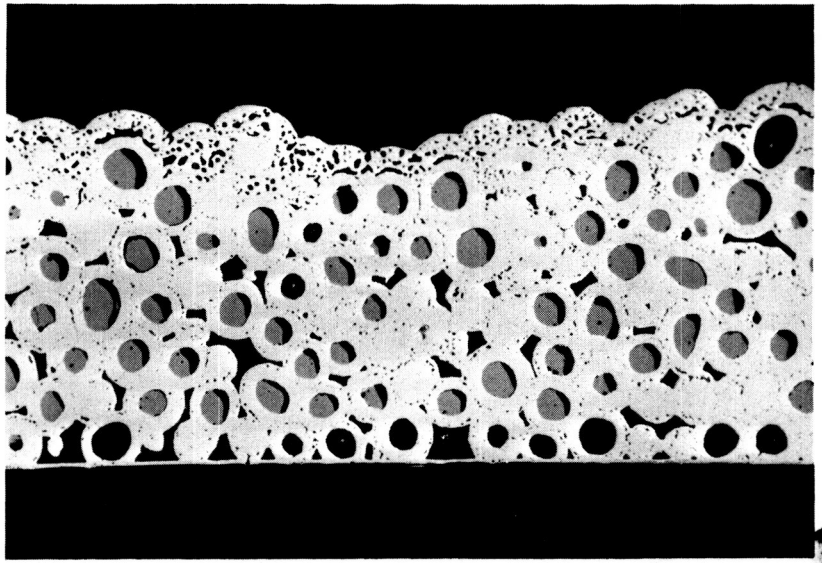


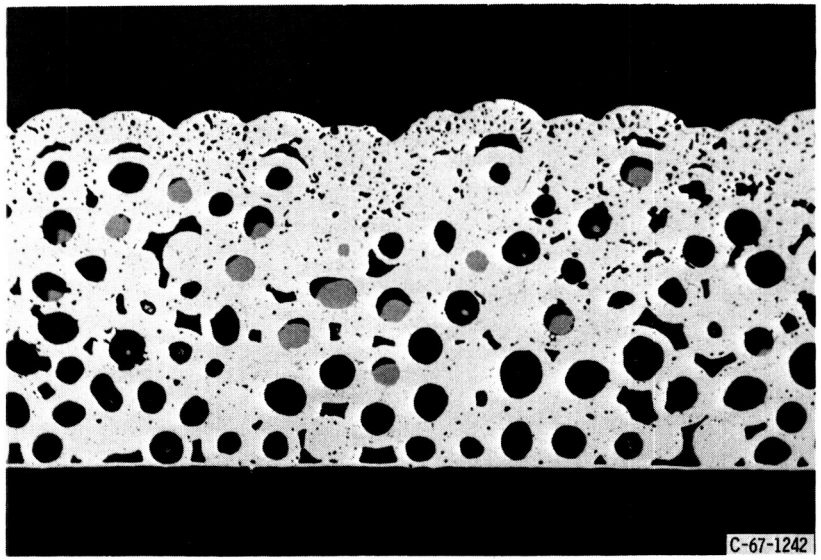
Figure 7. - Effect of thermal cycling on fuel loss from tungsten - 20-volume-percent uranium dioxide composites.



(a) Vapor-cemented specimen NU-11 after 25 cycles (typical of specimens showing poor cyclic-fuel-retention characteristics).

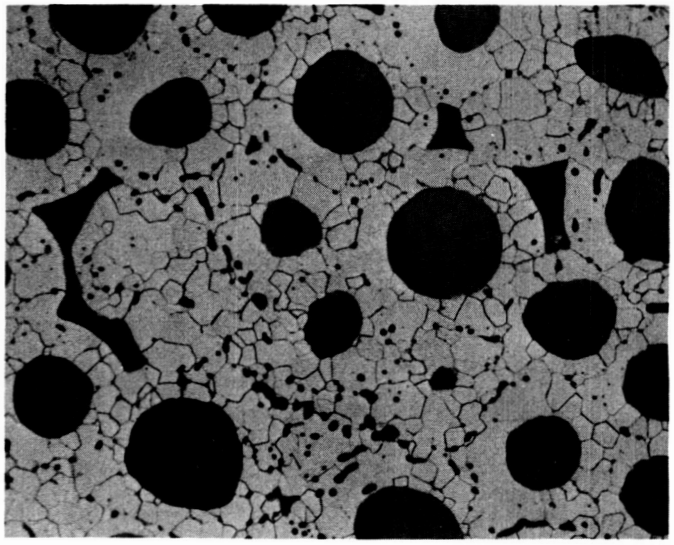


(b) Vapor-cemented specimen NU-1 after 25 cycles.

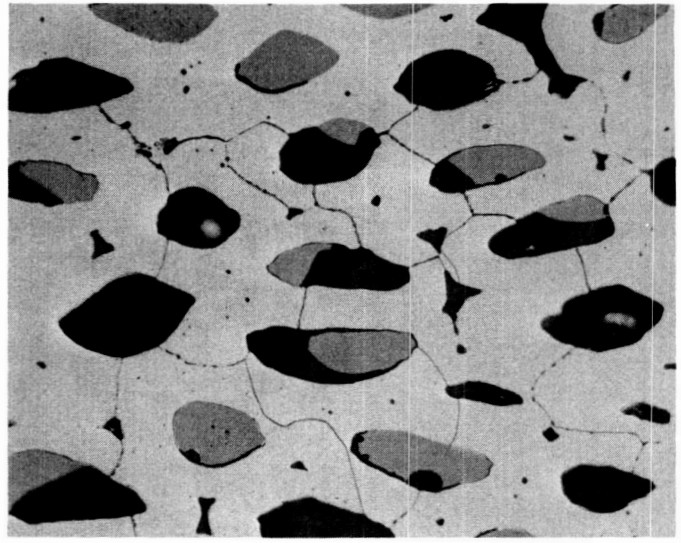


(c) Vapor-cemented specimen NU-1 after 80 cycles.

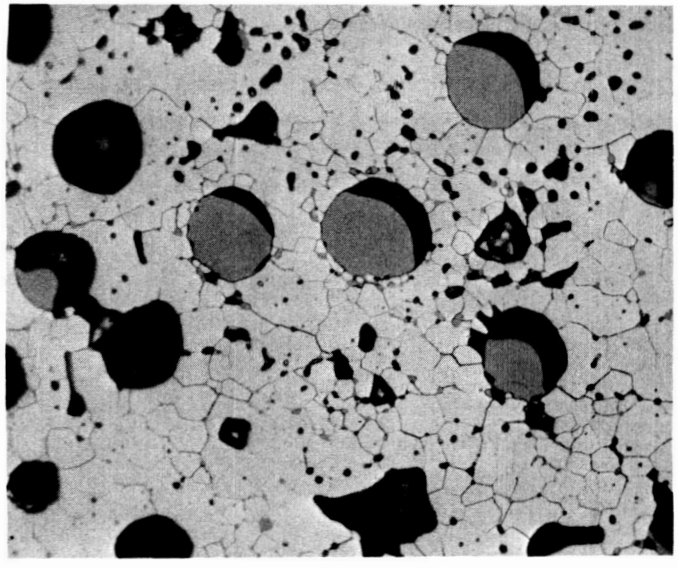
Figure 8. - Representative microstructures of tungsten - 20-volume-percent uranium dioxide after thermal cyclic testing in flowing hydrogen at 2500° C. Unetched. X100.



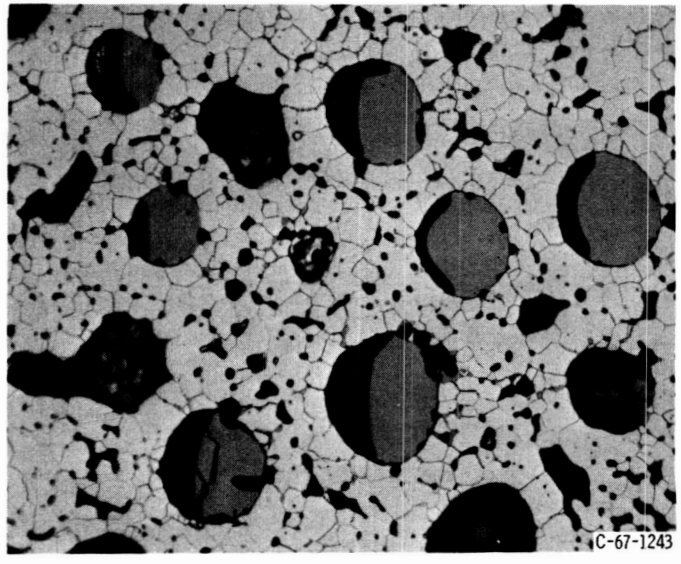
(a) Specimen NU-1 after 25 cycles.



(b) Roll-compacted, reference material after 10 cycles.



(c) Specimen NU-1 after 80 cycles.



(d) Specimen NU-11 after 25 cycles (typical of specimens showing poor cyclic-fuel-retention characteristics).

Figure 9. - Representative microstructures of tungsten - 20-volume-percent uranium dioxide composites after thermal cyclic testing in flowing hydrogen at 2500° C. Etchant, Murakami's reagent. X250.

[redacted]

"The aeronautical and space activities of the United States shall be conducted so as to contribute . . . to the expansion of human knowledge of phenomena in the atmosphere and space. The Administration shall provide for the widest practicable and appropriate dissemination of information concerning its activities and the results thereof."

—NATIONAL AERONAUTICS AND SPACE ACT OF 1958

NASA SCIENTIFIC AND TECHNICAL PUBLICATIONS

TECHNICAL REPORTS: Scientific and technical information considered important, complete, and a lasting contribution to existing knowledge.

TECHNICAL NOTES: Information less broad in scope but nevertheless of importance as a contribution to existing knowledge.

TECHNICAL MEMORANDUMS: Information receiving limited distribution because of preliminary data, security classification, or other reasons.

CONTRACTOR REPORTS: Scientific and technical information generated under a NASA contract or grant and considered an important contribution to existing knowledge.

TECHNICAL TRANSLATIONS: Information published in a foreign language considered to merit NASA distribution in English.

SPECIAL PUBLICATIONS: Information derived from or of value to NASA activities. Publications include conference proceedings, monographs, data compilations, handbooks, sourcebooks, and special bibliographies.

TECHNOLOGY UTILIZATION PUBLICATIONS: Information on technology used by NASA that may be of particular interest in commercial and other non-aerospace applications. Publications include Tech Briefs, Technology Utilization Reports and Notes, and Technology Surveys.

Details on the availability of these publications may be obtained from:

SCIENTIFIC AND TECHNICAL INFORMATION DIVISION

NATIONAL AERONAUTICS AND SPACE ADMINISTRATION

Washington, D.C. 20546

[redacted]